

WO 2005/026705 A1



---

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

## GAS DETECTION METHOD AND GAS DETECTOR DEVICE

The present invention concerns in particular low-cost infrared (IR) gas detection. A standard technology in this field consists in a thermal IR light source, an interference line filter, a sample chamber and an IR detector. The line filter corresponds to the characteristic absorption wavelength of the gas to be detected so  
5 that only light of this specific wavelength is incident onto the detector. If a gas to be detected is present in the sample chamber, part of the light is absorbed by the gas and the detector signal is lowered subsequently. In order to take into account the intensity variation of the light source due to aging, moisture or dirt, a part of the emitted light is directed outside the sample chamber onto a reference detector (so-  
10 called two-beam or reference-beam technique).

Such non-diffractive IR (NDIR) gas detectors suffer from two drawbacks. Firstly, thermal light sources have a high power consumption and a low light efficiency which makes battery-driven operation difficult and implies cooling issues. Secondly, the center wavelength of interference line filters is dependent on the temperature so  
15 that for different ambient temperatures, the detection operates at different positions of the gas absorption peak which in turn makes calibration difficult.

Recent developments with Vertical Cavity Surface Emitting Lasers (VCSEL) have shown a way to improve low-cost single gas detectors. VCSEL wavelengths are precisely defined and can be tuned over a few nanometers by a change of the VCSEL  
20 drive current. Such VCSEL diodes are meanwhile available for the near infrared (NIR) wavelength range of 1.3-2.05  $\mu\text{m}$ . Many of the gases detected by IR absorption have the first or second overtones of their absorption peaks in this wavelength range. Although these overtones are substantially weaker than the fundamental peaks, gas detection is very sensitive as VCSELs typically supply about 1000 times more light  
25 intensity than a thermal light source. An important advantage of VCSELs is their low power consumption of a few Milliwatt compared to a few Watt for thermal light sources.

A main difference between a standard NDIR detection and detection based on VCSELs is that NDIR techniques have a low spectral resolution and therefore  
30 measure gas absorption peaks which are typically several 100 nm wide. These broad absorption peaks are in fact composed of a large number of very sharp absorption lines. VCSELs emit with a very sharp wavelength peak which can be modulated within a few nanometers. For this reason, a VCSEL-based gas detector measures one single absorption line instead of a broad absorption peak.

Several authors have described a gas detection set up with a VCSEL source where the wavelength of the VCSEL is scanned across the absorption line of the gas as represented in figure 2. This scanning is done with a given modulation frequency  $F$ . This modulation is achieved by imposing a small alternating current (100  $\mu$ A typically) of frequency  $F$  onto a constant current above the lasing threshold (some mA typically). For some measurement techniques, this "constant current" is slowly swept across the whole operation range of the VCSEL in order to detect subsequent absorption lines. With such a set up, a line filter is no longer needed which is an important cost reduction factor for low-cost products.

10       The present invention is based on a source formed by a wavelength modulated VCSEL and uses the fact that the modulation of the wavelength is directly connected to a modulation of the VCSEL output intensity. The intensity of the light having passed the gas volume and being incident on the detector therefore shows a first modulation related to the VCSEL intensity and a second modulation related to the gas absorption as the wavelength is scanned across the gas absorption line.

15       With a standard IR detector which delivers a signal proportional to the incident radiation, the signal treatment consists in measuring the detector signal by a lock-in technique on twice the modulation frequency (2F-detection). By this, the DC signal component – which stems from the offset light detected throughout the modulation range – is suppressed. However, a reference beam has still to be used in order to obtain information about the overall light intensity of the initial light beam provided by the source for obtaining a precise value of the gas concentration. This reference beam is usually detected by a second specific detector. Thus, the generation and the detection of a reference beam complicate the device and increase its production cost.

25       An object of the present invention is to provide an efficient gas concentration measuring device or detector at low cost. In particular, the aim of the present invention is to solve the above mentioned problem relative to the reference beam.

30       Therefore, a first embodiment of the present invention concerns a gas detector device comprising a wavelength modulated laser source and a light sensor respectively arranged at the periphery of a detection region intended for receiving at least a gas the concentration of which is to be determined, said source providing an initial light signal which is wavelength modulated at a given frequency around an absorption line of said gas, said light sensor receiving a resulting light signal formed by the initial light signal having passed through said detection region, wherein the light sensor is of the type providing a detection signal substantially proportional to the time derivate of said resulting light signal thus forming an electronic signal which is substantially proportional to the time derivate of said resulting light signal, said device

further comprising first means for generating a first modulation reference signal at said given frequency and second means for generating a second modulation reference signal at twice this frequency, said electronic signal being multiplied by said first modulation reference signal and then integrated over time in order to provide a first measuring signal which is a function of the intensity of said initial light signal and substantially independent of the concentration of said gas, said electronic signal being also multiplied by said second modulation reference signal and then integrated over time in order to provide a second measuring signal which is a function of the gas absorption and substantially independent of an intensity modulation of said initial light signal at said given first frequency.

Further, a second embodiment of the present invention concerns a gas detector device comprising a wavelength modulated laser source and a light sensor respectively arranged at the periphery of a detection region intended for receiving at least a gas the concentration of which is to be determined, said source providing an initial light signal which is wavelength modulated at a given frequency around an absorption line of said gas, said light sensor receiving a resulting light signal formed by the initial light signal having passed through said detection region, wherein the light sensor is of the type providing a detection signal proportional to said resulting light signal, the device further comprising an electronic time derivator to which the detection signal is provided, this electronic time derivator generating an electronic signal which is substantially proportional to the time derivate of said resulting light signal, said device further comprising first means for generating a first modulation reference signal at said given frequency and second means for generating a second modulation reference signal at twice this frequency, said electronic signal being multiplied by said first modulation reference signal and then integrated over time in order to provide a first measuring signal which is a function of the intensity of said initial light signal and substantially independent of the concentration of said gas, said electronic signal being also multiplied by said second modulation reference signal and then integrated over time in order to provide a second measuring signal which is a function of the gas absorption and substantially independent of an intensity modulation of said initial light signal at said given frequency.

Owing to the features of the gas detector device of the invention, only a single sensor unit is needed for one laser source, all necessary information for determining a precise gas concentration value being given by the processing of the generated electronic signal which is proportional to the derivate of the light signal received by the sensor unit after having passed through a sample of the defined gas.

Other particular features and advantages of the present invention will be described with reference to the following description and the annexed drawings, given by way of non limiting embodiments, in which:

- 5       - Figure 1 shows schematically a gas detector device according to the present invention;
- Figure 2 shows the wavelength modulation of a VCSEL source around a gas absorption line used to detect the concentration of this gas;
- Figure 3 shows the light intensity modulation of the initial light beam provided by the VCSEL source and resulting from the wavelength modulation of figure 2;
- 10       - Figure 4 gives the graph of a resulting light beam having passed through a gas sample;
- Figure 5 is a graph of a signal proportional to the time derivate of the signal shown on figure 4;
- Figures 6 and 7 respectively show first and second modulation reference
- 15       signals at frequencies  $F$  and  $2F$ ,  $F$  being the frequency of the wavelength modulation given on Figure 2;
- Figure 8 shows a first resulting signal generated by the multiplication of the signal of figure 5 by the first modulation reference of figure 6;
- Figure 9 shows a second resulting signal generated by the multiplication of
- 20       the signal of figure 5 by the second modulation reference of figure 7;
- Figures 10 and 11 show variations in a first measuring signal and in a second measuring signal provided by a gas detector device of the present invention in function of the central wavelength of the modulation of the VCSEL source relative to an absorption line;
- 25       - Figure 12 shows variations in the second measuring signal in function of the amplitude of the VCSEL modulation;
- Figure 13 is a schematic view of a first embodiment of the gas detector device according to the present invention; and
- Figure 14 is a schematic view of a second embodiment of the gas detector
- 30       device according to the present invention.

With reference to figures 1 to 9, the method for detecting a gas concentration according to the present invention will be described.

As schematically shown in Figure 1, the gas detector device according to the invention comprises a light source formed by a VCSEL 2, a sample chamber or

35       detection region 4 where a gas to be detected can be introduced, a light detector 6 and processing means 8 which provide two measuring signal  $S_{MF}$  and  $S_{M2F}$  allowing a gas concentration to be defined. The VCSEL generates an initial light beam  $S_0$  which

is wavelength modulated. This light beam passes through the region 4. Due to the gas absorption, the initial light signal presents an intensity variation after having passed through the gas detection region 4 and thus the detector 6 receives a resulting light signal  $S_G$ . The detector provides a corresponding detection signal  $S_D$  to the processing means 8.

As shown in Figure 2, the VCSEL wavelength  $\lambda$  (center of the light intensity peak 10) is modulated within a small range around a given gas absorption line 12. This wavelength modulation is directly coupled to an amplitude modulation of the initial light intensity, indicated in figure 2 by different intensity peak heights. Figure 3 shows the intensity variation of the initial light signal  $S_0$  over time resulting from an alternative scanning around the gas absorption line.

The intensity variation of the resulting light signal  $S_G$  exiting the gas absorption chamber or region 4 is shown in Figure 4. This signal  $S_G$  has therefore two contributions:

- The first contribution stems from the fact that the intensity of the VCSEL varies (approximately linearly) with its wavelength. This contribution is independent of the gas absorption and exists even if no gas is present.
- The second contribution stems from the gas absorption as the wavelength is scanned across the gas absorption line. This contribution is linearly proportional to the light intensity emitted by the VCSEL and it is a function of the gas concentration in the gas absorption region.

To separate these two contributions, the measurement principle of the present invention first proposes to obtain the time derivate of the resulting light signal  $S_G$  and then to process the time derivate signal 18 shown in Figure 5 with so-called lock-in amplifiers as will be described in more details hereafter.

In a lock-in amplifier, an modulated signal is multiplied with a symmetrical rectangular signal ("modulation reference") which has a well defined phase relation to the modulated signal. The resulting electronic signal is then integrated over a number of modulation periods in order to give a measuring signal at the output of the lock-in amplifier.

Figure 6 shows a first modulation reference signal 20 at the Frequency  $F$  which corresponds to the Frequency of the scanning by the VCSEL source, that is to the Frequency of the intensity modulation 22 of the initial light signal  $S_0$ . This figure 6 also shows the phase relation between the intensity modulation signal 22 and the first modulation reference 20 generated from this signal 22. Figure 7 shows a second modulation reference signal 24 at twice said Frequency  $F$ . This figure 7 also shows

the phase relation between the intensity modulation signal 22 and the second modulation reference 24 generated from this signal 22.

According to the invention, the time derivate of the resulting light signal  $S_G$  is either directly obtained by the use of a pyroelectric sensor which yields a signal  
5 substantially proportional to the change of the light intensity received by this pyroelectric sensor or it is obtained by an electronic derivator in the case that the employed sensor produces a signal  $S_D$  substantially proportional to the incident light signal  $S_G$  (i.e. photodiode, thermoelement, bolometer).

Figure 8 shows the resulting curve 26 of the multiplication of the time derivate  
10 intensity signal 18 (Figure 5) with the first modulation reference signal 20 at the VCSEL modulation frequency  $F$ . It is obvious that the subsequently positive and negative contributions from gas absorption cancel out in an time integration of the resulting curve 26, so called F-detection. The result of such a time integration is a first measuring signal  $S_{MF}$  that is a function of the modulation of the VCSEL intensity and  
15 related to the overall VCSEL intensity, but which is independent of the presence of a gas in the detection region.

Figure 9 shows the resulting curve 28 of the multiplication of the time derivate intensity signal 18 (Figure 5) with the second modulation reference signal 24 at twice  
20 said frequency  $F$ . Here, the contribution from the VCSEL intensity modulation cancel out in a time integration of the resulting curve 28, so called 2F-detection, whereas the individual contributions of the gas absorption will add up. The result of such an integration is a second measuring signal  $S_{M2F}$  that is a function of the gas absorption and thus of the gas concentration. Said Integration cancels the contribution which is independent of gas absorption.

25 The second measuring signal  $S_{M2F}$  is in fact substantially proportional to the overall light intensity coming from the VCSEL. By dividing this second measuring signal  $S_{M2F}$  by the first measuring signal  $S_{MF}$ , one obtains a value which is a function of the gas concentration but independent of the light intensity incident onto the detector.

A gas sensor according to the invention, based on a VCSEL and a two-channel  
30 lock-in amplifier, provides therefore gas absorption signal and VCSEL intensity reference with one single detector so that the need for a separate physical reference channel as used in conventional NDIR sensors is suppressed. Moreover, the intensity reference value is obtained directly from the light incident on the detector whereas a two-beam NDIR sensor obtains such a reference from a separate beam which may  
35 not give information about changes in the measurement beam (i.e. aging of optical components or misalignments due to thermal variations).

The analysis of the measuring signals  $S_{MF}$  and  $S_{M2F}$  shows that the phase relation between the intensity modulation signal of the VCSEL and the modulation reference signals 20 and 24 as depicted in Figures 6 and 7 is critical for the measurement principle. A deviation from this given phase relation will result in contributions of the gas absorption signal ( $S_{M2F}$ ) to the intensity reference signal ( $S_{MF}$ ), and vice versa.

A more extensive analysis of the signals shows that the signal processing described above is not dependent on the shape of the VCSEL AC modulation, i.e. instead of the triangular modulation depicted in Figure 3, the modulation may as well be sinusoidal, saw tooth or of another shape.

VCSEL sources can be wavelength modulated in a wide frequency range from a few Hz to several MHz. As a consequence, a gas sensor according to the present invention can be built to yield response times from several seconds to several microseconds, depending on the required specification.

Figures 10 and 11 show the influence of the position of the VCSEL central wavelength (which is adjusted by the VCSEL DC current) with respect to the center of gas absorption line on the two measuring signals. The curves were taken by ramping the VCSEL DC current up while applying a small AC modulation corresponding to a wavelength modulation of 0.15 nm. It is evident that the measurement principle as described above only holds if the VCSEL wavelength is exactly centered on the gas absorption line and the AC modulation scans symmetrically across the gas absorption line. A deviation from this center position yields a decreased absorption signal as well as an error in the reference signal. However, the latter decreases with decreasing gas concentration.

As shown in Figure 12, the amplitude of the VCSEL AC modulation has an influence on both first and second measuring signals. The signal analysis shows that the gas absorption signal  $S_{M2F}$  has a maximum for a modulation amplitude on the order of magnitude of the width of the gas absorption line (0.1-0.15 nm). The error of the intensity reference signal  $S_{MF}$  decreases with increasing modulation amplitude. In consequence, the modulation amplitude can be optimised for a given specification of the gas sensor.

Since the wavelength of a VCSEL is a function of ambient temperature, the center wavelength of the VCSEL has to be kept locked onto the exact wavelength of the gas absorption line (see Figures 10 and 11). This can be achieved by including a sealed transparent cell in the light path which contains the gas to be detected. On switching on of the gas sensor, the VCSEL DC current is slowly ramped up from a default DC value while scanning with the AC frequency  $F$  until the gas absorption line



comes to lie within the AC modulation range. From this point on, the gas absorption signal will be non-zero and a feedback loop to the DC current source will keep this signal at its maximum which corresponds to the locking of the VCSEL central wavelength to the gas absorption line center. However, because the wavelength variation of a given VCSEL is limited, it is necessary to keep the VCSEL source approximately at a predefined temperature. In the case of a CO<sub>2</sub> detector for ambient air, the sealed cell can be omitted as the natural concentration of CO<sub>2</sub> of 350-400 ppm is sufficiently high for the described locking purpose.

The fact that the light emitted by a VCSEL is highly directional allows for a simple design for a multi-gas sensor without further optics. In such a device, several VCSELs (each of a wavelength corresponding to a different gas) are mounted into a laser head whereas the detector is an array of as many light sensors as the laser head contains VCSELs. Mounting is done in a way that the laser beam of each VCSEL aims at a different sensor which yields a very compact multi-gas detection device for two, three or more different gases.

Concerning the laser source, a distributed feedback laser (DFBLaser) can also be selected in the frame of the present invention. VESELS and DFBLasers are preferred laser sources.

Figure 13 shows a first embodiment of a gas detector device according to the invention. This device comprises a laser light emitting head 32 in which two VCSEL sources 34 and 36 are arranged. Thus, this device forms a detector for two different gases, both sources being respectively chosen for corresponding to selected absorption lines of these two gases. This head 32 further comprises a sealed cell filled with said two different gases for precisely determined the electrical current value to be furnished to each source 34 and 36 so that the central wavelength of the provided light peak corresponds to the center of the absorption line of the respective gas, as explained here-before. Finally the head 32 comprises a temperature sensor 40 electrically connected to power supply means 42 of heating means 44 located in the region where the sources are arranged.

The gas detector device has a sample chamber or gas detection region 48 through which the two laser beams 50 and 52 provided by the two laser sources pass through. The two laser beams are then received by two respective light sensors 54 and 56 arranged on a common base 58. In this first embodiment, the two sensors are of the type providing an electrical detection signal substantially proportional to the incident light signal on the sensor, as a thermoelement or a bolometer or preferably a photodiode. According to the invention, the two sensors 54 and 56 are connected, through an electronic selector 62, to an electronic time derivator 64. This derivator

thus provides an electronic signal which is substantially proportional to the time derivate of said incident light signal to preamplifier means 66.

The gas detector device further comprises supply control means 70 connected to electrical supply means 72 which furnish an electrical current to the sources 34 and 5 36 through an electronic selector 74. The supply control means 70 have a first part 76 for defining a DC current signal and a second part 78 for defining an AC current signal at a given reference frequency F generating an alternative scanning around the gas absorption line as explained before. The processing means of the device also comprise first means 80 for generating a first modulation reference signal at said 10 reference frequency F and second means 82 for generating a second modulation reference signal at twice said reference frequency F. According to the method of the present invention described before, these first and second modulation reference signals are respectively provided to two lock-in amplifiers 84 and 86 in which these reference signals are respectively multiplied with the signal provided by the time 15 derivator 64 to these two lock-in amplifiers through the preamplifier means, and then integrated over several time periods of the first modulation reference signal. The first lock-in amplifier 84 provides a first measuring signal which is independent from the gas absorption as explained before. The second lock-in amplifier 86 provides a second measuring signal which is independent of the modulation of the initial light 20 signal generated by the respective source and relative to the gas absorption and thus to the gas concentration in the region 48.

In a preliminary step, the second measuring signal is used to define the DC current signal by detecting the maximum of this second measuring signal when the DC current level is linearly varied. It is to be noted that this preliminary step can be 25 avoided if the device is equipped with a very precise temperature control for the laser source.

Finally, the second measuring signal is divided by the first measuring signal in an processing unit 90 in which the result of this division is further processed in order to furnish a useful signal or information relative to the presence of a given gas or to its 30 concentration.

Figure 14 shows a second embodiment of a gas detector device according to the invention. The references already described in the first embodiment will not be described again here. This second embodiment differentiates from the first one in that the two light sensors 94 and 96 are of a specific type and provide directly an electrical 35 detection signal which is substantially proportional to the time derivate of the incident light signal on these sensors. Preferably, the sensors 94 and 96 are pyroelectric

sensors. Thus the electronic time derivator is no more needed in this second embodiment. The electrical detection signal is directly provided to the two lock-in amplifiers 84 and 86 through a preamplifier 66.

5           In a preferred embodiment of the device of the invention, both source and light sensor are located on the same side of the gas detection region, a reflective structure being arranged at the opposite side. Thus, for a given length of the gas detection region, the path of the light through the gas sample is twice as long as in the first and second embodiments shown on Figures 13 and 14. Further, the source, the sensor  
10           and the electronic elements can be integrated in/on a common substrate, which is very advantageous and cost reducing. The reflective structure can be used for focusing the light beam, especially when its numerical aperture is relatively high.

          In another embodiment of the invention for the detection of two gases, the device comprises two laser sources but only a single light sensor, the two generated  
15           light beams being oriented in order to be incident on this light sensor. Like in the embodiment of Figures 13 and 14, a time multiplexing in the control of both sources allows to measure the concentration of two gases. Thus the two light beams are alternatively directed on the single light sensor.

          Finally, if the absorption lines of different gases are sufficiently narrow, it is  
20           possible to use only one laser source for detecting these gases.

          The gas detector according to different features of the invention has the following advantages:

- Suppression of the reference beam which is especially important for multi-gas measurements,
- 25           - No influence of the degrading of optical components or VCSEL intensity,
- Low power consumption, enabling wireless devices,
- Low heat dissipation, thus no cooling issues,
- Temporal resolution down to microseconds,
- Automatic detection of VCSEL malfunction,
- 30           - Active temperature compensation,
- Spectral auto-locking,
- Compact design for multi-gas detector,
- Low fabrication cost for large series as VCSELs, detector and readout electronics can all be fabricated by batch processing techniques.

CLAIMS

1. A gas detector device comprising a wavelength modulated laser source (2; 34, 36) and a light sensor (94, 96) respectively arranged at the periphery of a detection region (48) intended for receiving at least a first gas the concentration or presence of which is to be determined, said source providing an initial light signal  $S_0$  which is wavelength modulated at a first frequency  $F$  around an absorption line of said first gas, said sensor receiving a resulting light signal ( $S_G$ ) formed by the initial light signal having passed through said detection region, characterized in that the light sensor is of the type providing a detection signal substantially proportional to the time derivate of said resulting light signal thus forming an electronic signal which is substantially proportional to the time derivate of said resulting light signal, said device further comprising first means (80) for generating a first modulation reference signal (20) at said first frequency and second means (82) for generating a second modulation reference signal (24) at twice said first frequency, said electronic signal being multiplied by said first modulation reference signal and then integrated over time in order to provide a first measuring signal ( $S_{MF}$ ) which is a function of the intensity of said initial light signal and substantially independent of the concentration of said first gas, said electronic signal being also multiplied by said second modulation reference signal and then integrated over time in order to provide a second measuring signal ( $S_{M2F}$ ) which is a function of the first gas absorption and substantially independent of an intensity modulation of said initial light signal at said first frequency.

2. A gas detector device comprising a wavelength modulated laser source (2; 34, 36) and a light sensor (54, 56) respectively arranged at the periphery of a detection region (48) intended for receiving at least a first gas the concentration or presence of which is to be determined, said source providing an initial light signal ( $S_0$ ) which is wavelength modulated at a first frequency ( $F$ ) around an absorption line of said first gas, said light sensor receiving a resulting light signal ( $S_G$ ) formed by the initial light signal having passed through said detection region, characterized in that the light sensor is of the type providing a detection signal proportional to said resulting light signal, the device further comprising an electronic time derivator (64) to which the detection signal is provided, this electronic time derivator provided an electronic signal which is substantially proportional to the time derivate of said resulting light signal, said device further comprising first means (80) for generating a first modulation reference signal (20) at said first frequency and second means (82) for generating a second modulation reference signal (24) at twice said first frequency, said electronic signal being multiplied by said first modulation reference signal and then integrated over time in order to provide a first measuring signal ( $S_{MF}$ ) which is a function of the

intensity of said initial light signal and substantially independent of the concentration of said first gas, said electronic signal being also multiplied by said second modulation reference signal and then integrated over time in order to provide a second measuring signal ( $S_{M2F}$ ) which is a function of the first gas absorption and substantially

5 independent of an intensity modulation of said initial light signal at said first frequency.

3. Device according to claim 1 or 2, characterized in that it further comprises processing means (90) receiving said first and second measuring signals and providing an information relative to the concentration of the first gas.

4. Device according to claim 1, characterized in that said light sensor is  
10 formed by a pyroelectric sensor.

5. Device according to claim 1, characterized in that said light sensor is formed by at least one photodiode or one thermo-element or one bolometer.

6. Device according to any of the preceding claims, characterized in that said source is a vertical cavity surface emitting Laser (VCSEL).

15 7. Device according to any of claims 1 to 5, characterized in that said source is a distributed feedback laser (DFBL).

8. Device according to any of the preceding claims, characterized in that said laser source and said light sensor are both located at a same side of the detection region, a reflective structure being arranged at the other side of this  
20 detection region.

9. Device according to claim 8, characterized in that said laser source and said light sensor are both integrated in/on a common substrate.

10. Device according to any of the preceding claims, characterized in that it comprises at least two laser sources for detecting at least two different gases and a single light sensor on which both generated laser beams are alternatively directed in order to provide an information relative to each of both gases.

1 / 6

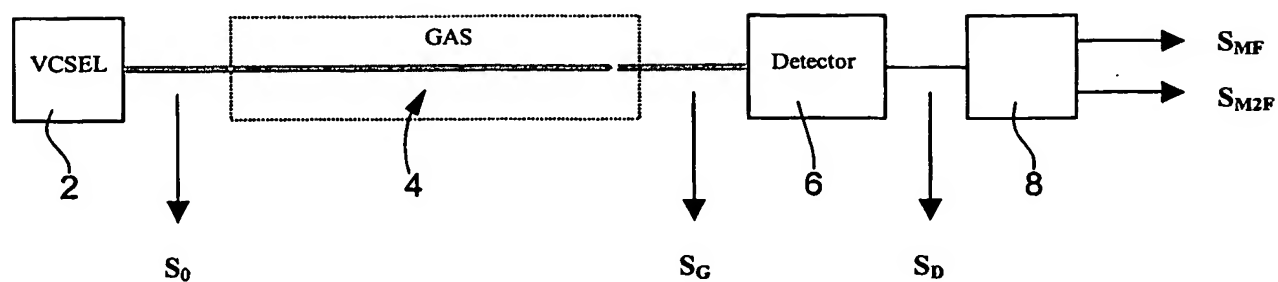


Fig. 1

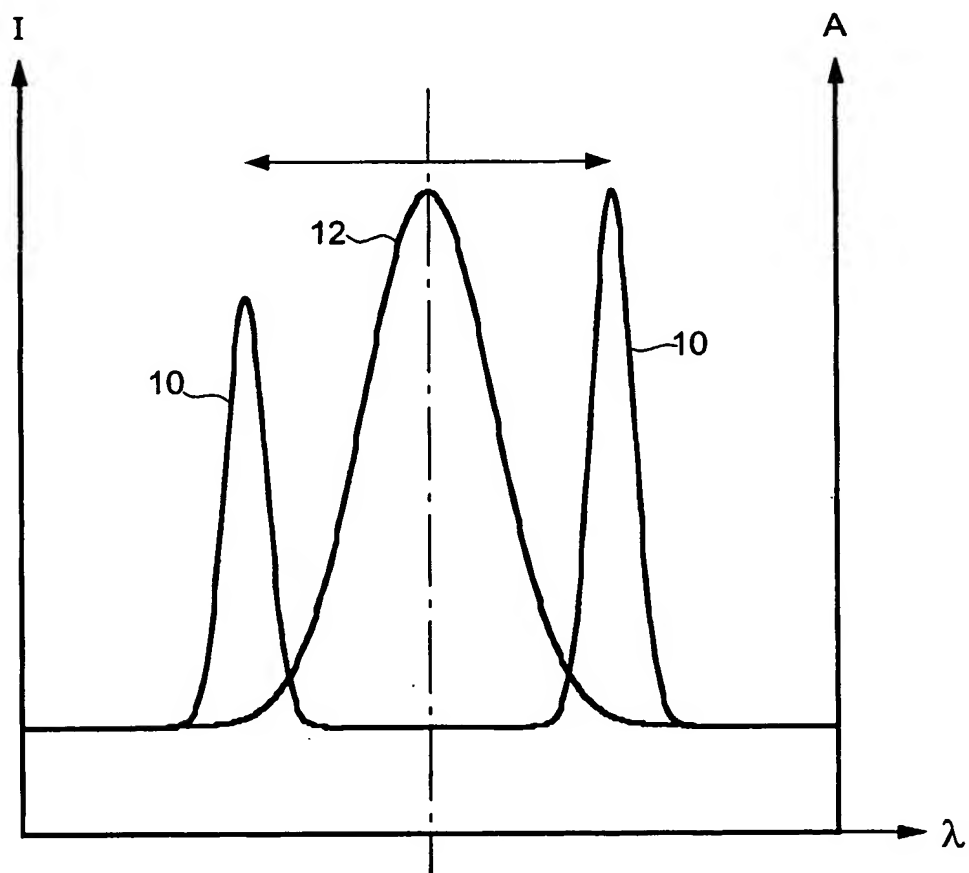


Fig. 2

2 / 6

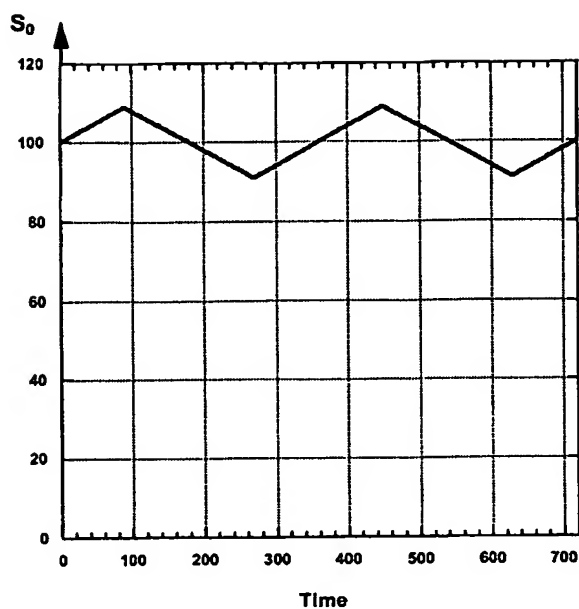


Fig. 3

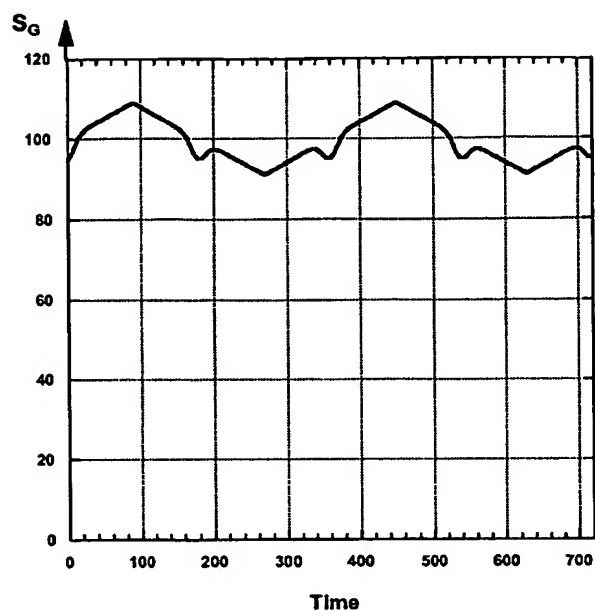


Fig. 4

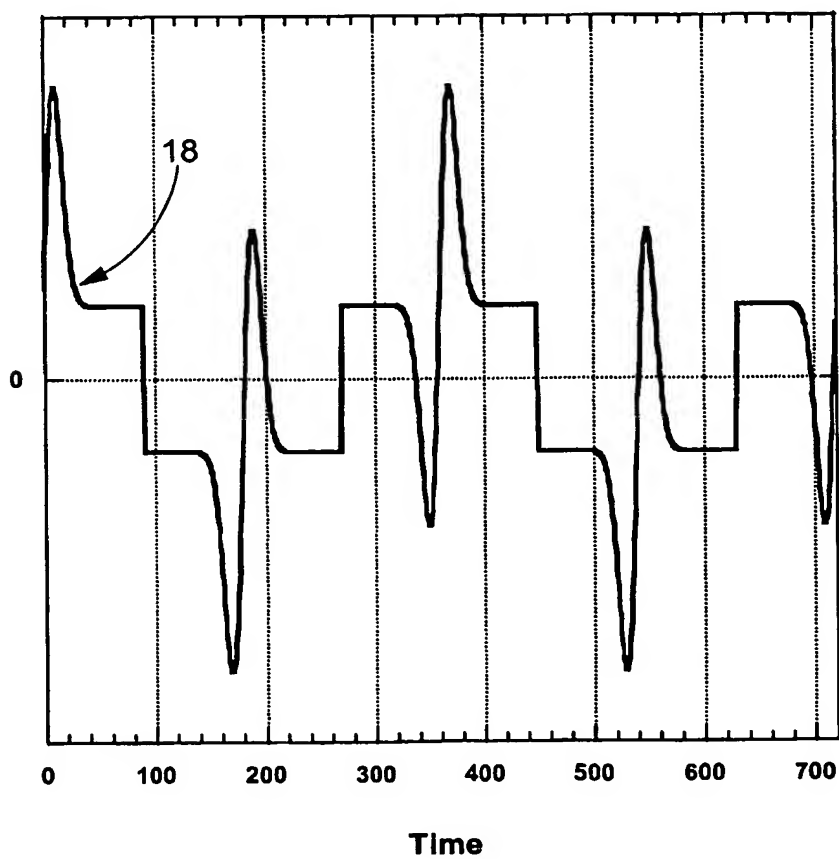


Fig. 5

3 / 6

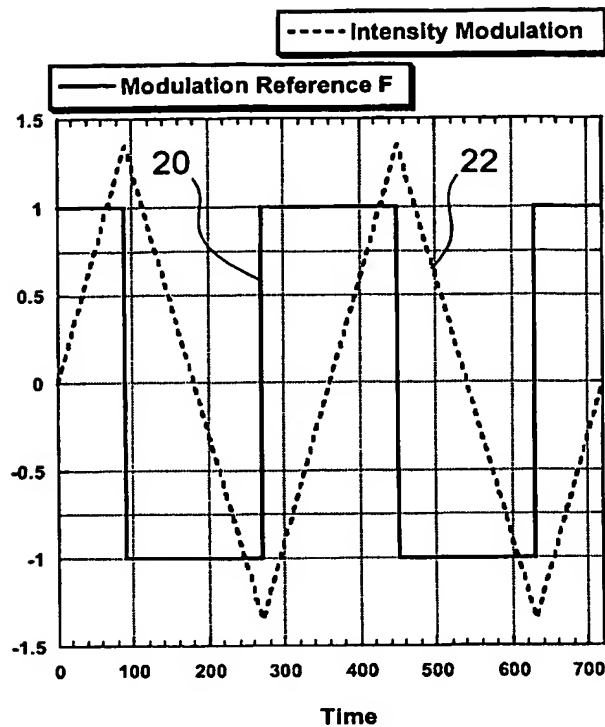


Fig. 6

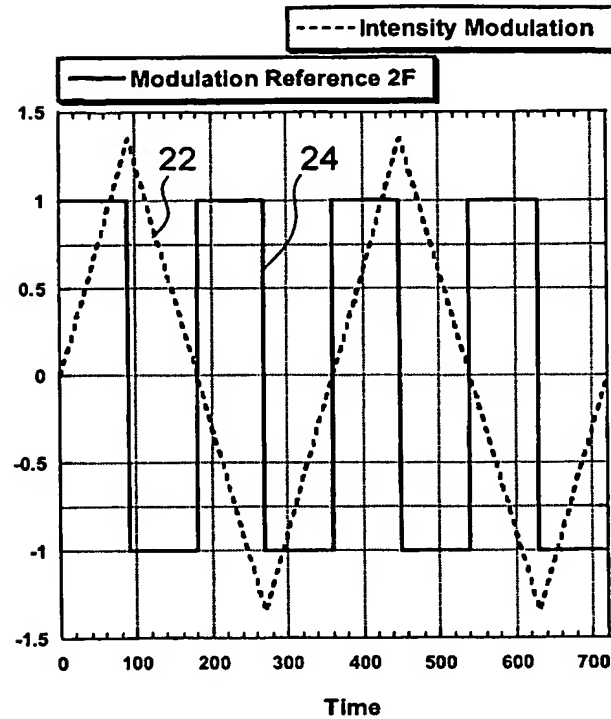


Fig. 7

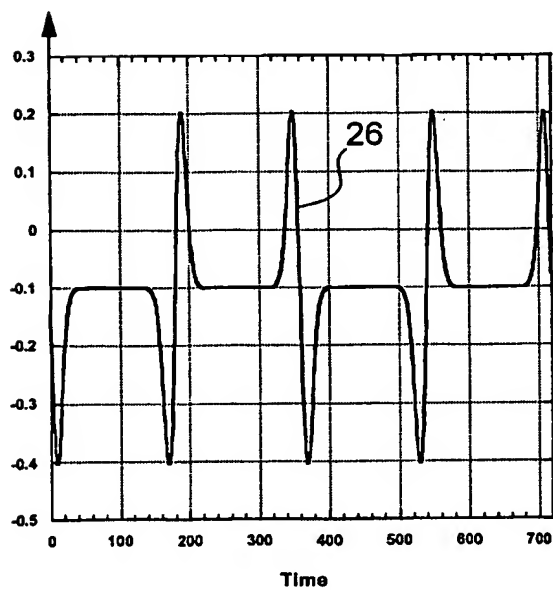


Fig. 8

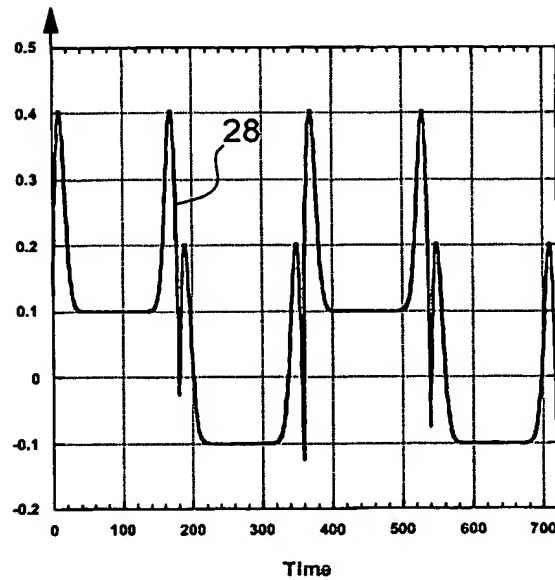


Fig. 9



4 / 6

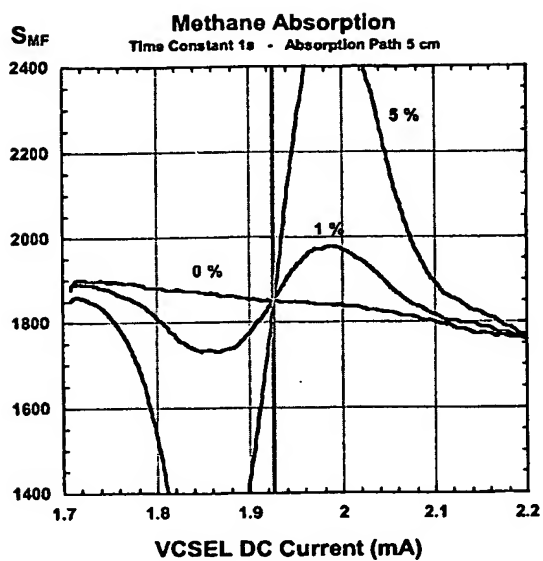


Fig. 10

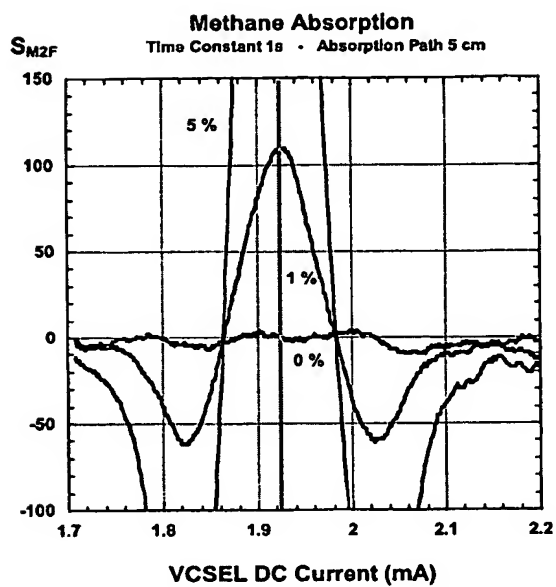


Fig. 11

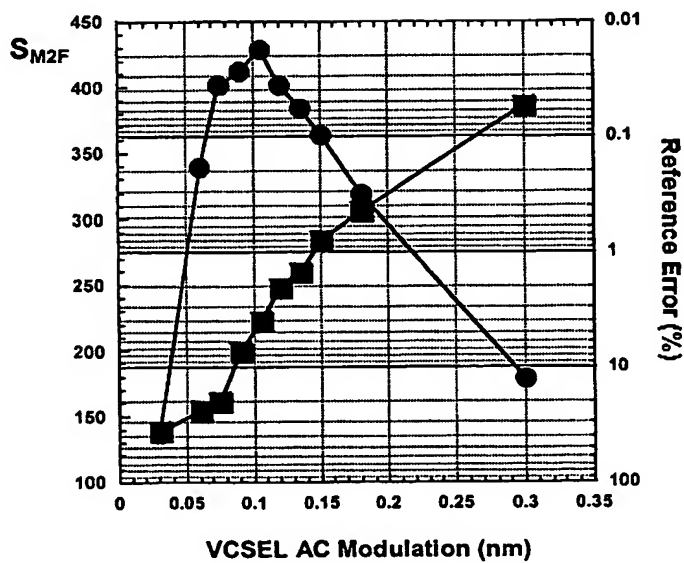


Fig. 12

5 / 6

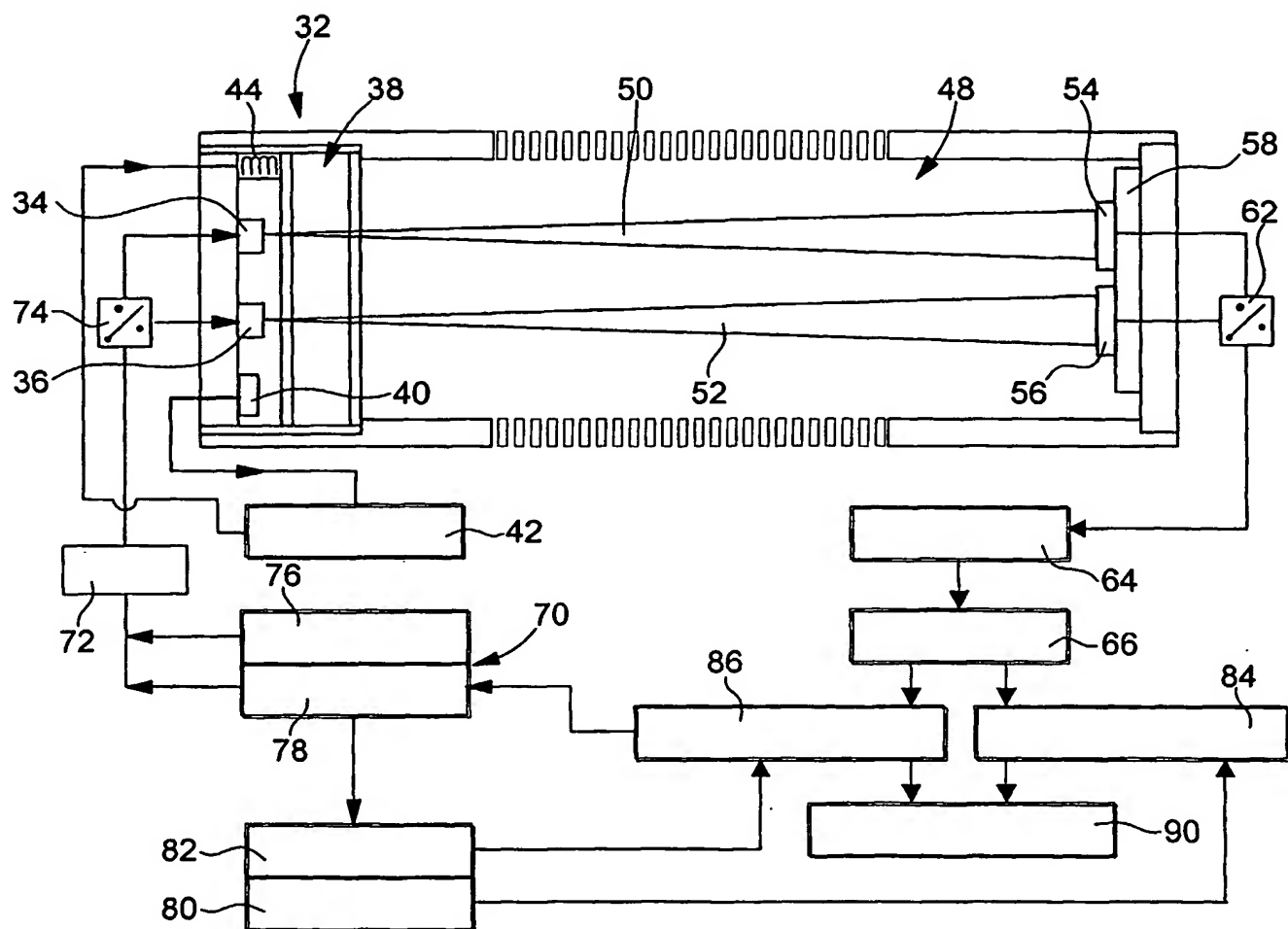


Fig. 13

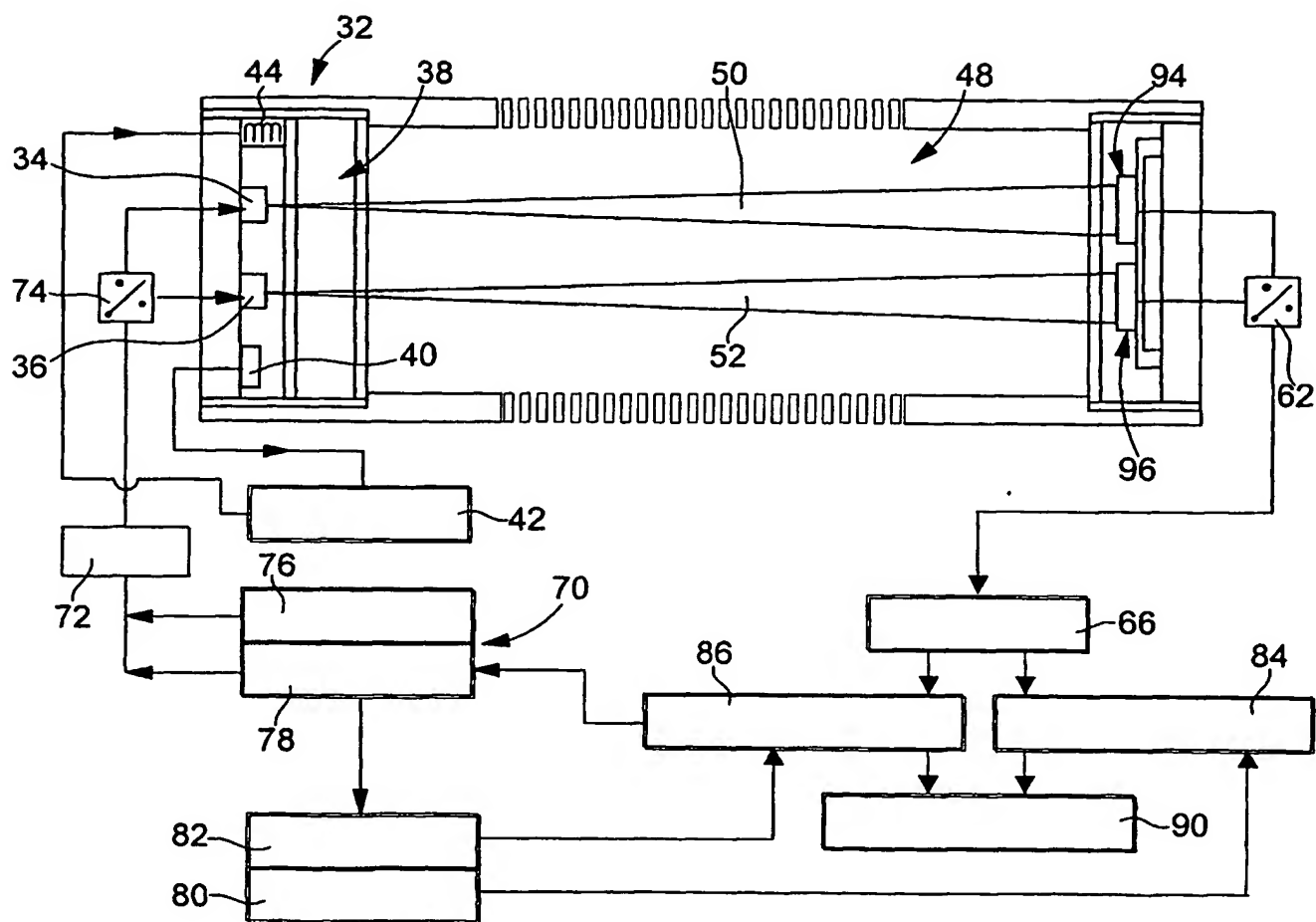


Fig. 14

# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/2004/008584

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 7 G01N21/35 G01N21/39

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 7 G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, WPI Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 6 356 350 B1 (BOMSE DAVID S ET AL) 12 March 2002 (2002-03-12)	1,2,6
A	column 6, line 47 - column 8, line 31; figure 1	3
Y	US 6 172 759 B1 (GOLDSTEIN MARK K) 9 January 2001 (2001-01-09)	1,2
Y	US 2003/081875 A1 (KOCHERGIN VLADIMIR ET AL) 1 May 2003 (2003-05-01)	1,2,6
A	paragraph '0036! - paragraph '0043! paragraph '0060!; claims 18,19	3,8
	----- -/--	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \*8\* document member of the same patent family

Date of the actual completion of the international search

21 December 2004

Date of mailing of the international search report

30/12/2004

Name and mailing address of the ISA  
European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Stuebner, B

# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/2004/008584

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 6 064 488 A (BIEN FRITZ ET AL) 16 May 2000 (2000-05-16)	1,2
A	column 2, line 40 - column 3, line 48 column 4, line 35 - column 5, line 33; figure 1	3,6,8
A	----- PATENT ABSTRACTS OF JAPAN vol. 2000, no. 20, 10 July 2001 (2001-07-10) & JP 2001 074654 A (MITSUBISHI HEAVY IND LTD), 23 March 2001 (2001-03-23) abstract -----	1,2

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/2004/008584

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 6356350	B1	12-03-2002	NONE
US 6172759	B1	09-01-2001	NONE
US 2003081875	A1	01-05-2003	EP 1444758 A1 11-08-2004 WO 03038953 A1 08-05-2003 US 2004202399 A1 14-10-2004
US 6064488	A	16-05-2000	NONE
JP 2001074654	A	23-03-2001	NONE